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審査請求 有

⑫カーテンレール

⑬実 願 昭46-5591

⑭出 願 昭46(1971)2月5日

⑮考 案 者 出願人と同じ

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実用新案登録請求の範囲

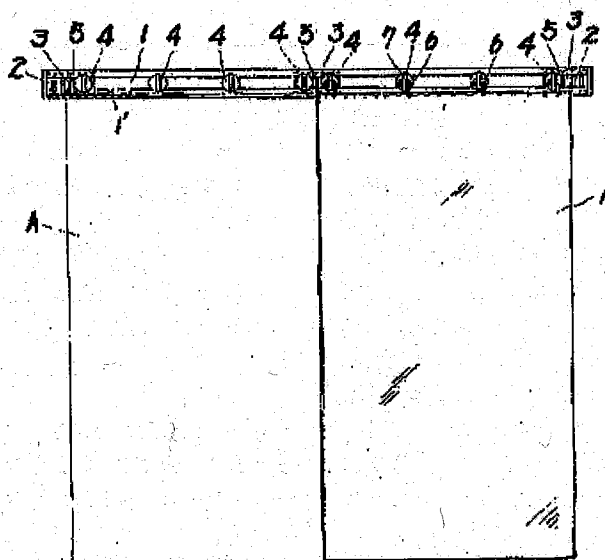
カーテンレール本体1の両端に設けたアンカー
胴体部2の内側に永久磁石体3を取付けるととも
に、これに対応する吸着軟鉄5を、カーテンレ
ール本体1内に挿嵌したカーテンランナー4群の両

端のカーテンランナー4に取付け、且カーテン幅
の合わせ端部となる対向のカーテンランナー4、
4に永久磁石体3と吸着軟鉄5を各々対応する如
く取付けしてなるカーテンレール。

図面の簡単な説明

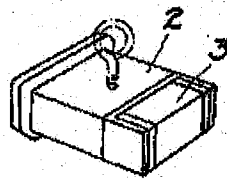
図面は本考案の実施例を示すもので、第1図は
使用状態を示す正面図、第2図はアンカー胴体部
の斜面図、第3図イ、ロはカーテンランナー部の
斜面図、第4図は同カーテンランナーの係合部
を示す断面図、第5図は永久磁石体の平面図であ
る。1……カーテンレール本体、2……アンカー
胴体部、3……永久磁石体、4……カーテンラン
ナー、5……吸着軟鉄。

第1図

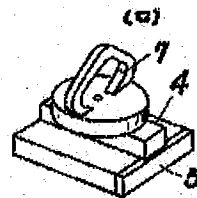
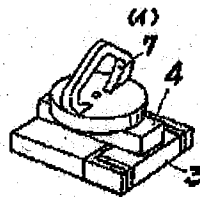


実開 昭47-5118(2)

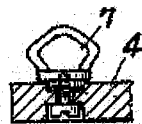
第2図



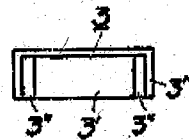
第3図



第4図



第5図



JP,47-005118,U

☒ STANDARD ☐ ZOOM-UP ROTATION No Rotation ☐ REVERSAL

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Translation of Utility Model Publication

Jpn. U.M. Publication No. 47-005118

Filing No.: 42-086109

Filing Date: October 10, 1967

Applicant: SHIMADZU CORP

Inventors: DATE GEN

SHIMATANI IKUJI

KOKOKU Date: February 22, 1972

Int.Cl.: G01 n

Title of the Invention

Sample contamination prevention apparatus in X-ray micro-analyzer

[Brief Description of the Drawing]

FIG. 1 is a view showing a sample contamination prevention apparatus in an X-ray micro-analyzer of the present invention;

FIG. 2 is an enlarged view of a liquid reservoir A of FIG. 1;

FIG. 3 is an enlarged view of a liquid feed pump section P of FIG. 1;

FIG. 4 is a graph showing a measurement result of contamination; and

Tables 1 and 2 show a part of the result shown in the graph of FIG. 4.

[Detailed Description of the Invention]

The present invention relates to a sample contamination prevention apparatus in an X-ray micro-analyzer that projects a finely focused electron beam onto the surface of an unknown sample so as to allow analysis of the distribution state of elements existing in a minute portion of the sample by observing an X-ray spectrum of a rational X-ray group generated from the minute portion of the sample.

In the X-ray micro-analyzer, when the sample is contaminated, unexpected variation occurs in the analysis result, and when an electron optical system is contaminated, the position of an electron probe is unexpectedly moved due to charging. Therefore, in order to prevent the contamination and to achieve a stable continuous analysis for a long time, it is necessary to set the inside of a vacuum vessel (mirror body) housing an X-ray spectroscopy system, electron optical system, and observation optical system in

a high vacuum state (about 2×10^{-5} mmHg).

In such apparatus, air evacuation is generally performed using an oil rotary pump and an oil diffusion pump in order to obtain a high vacuum and to shorten the time required for measurement. Further, additional rotary pump for preliminary evacuation is provided in a sample chamber and an electron gun section. As a result, there is a possibility that oil vapor or oil may flow backward in the mirror body and float therein. When an electron beam is irradiated on the sample in this state, an organic substance (esp. oil vapor) is decomposed to cause carbon to adhere to the high temperature portion of the electron beam irradiation surface, contaminating the sample. Note that it is accepted that the adherence of carbon to the sample surface, i.e., sample contamination by carbon occurs in a condition where the oil rotary pump and oil diffusion pump serving as an evacuation pump are not used. Further, although the cause of the carbon adherence is theoretically unknown at the moment, it is thought that an organic substance existing in the vacuum vessel is decomposed by the sample heated by electron impact followed by adherence to the sample surface.

Adverse effect of the contamination becomes pronounced in quantitative analysis of a light element and, especially, in quantitative analysis of an ultra-light element. That is, the rational X-ray from the light element is easily absorbed, so that it is absorbed by a contamination layer, i.e., a carbon layer formed on the sample surface and, further, rational X-ray of carbon is discharged from the carbon layer to cause variation in the analysis value, degrading reproducibility.

The present invention has been made in view of the above situation, and an object thereof is to provide a sample contamination prevention apparatus capable of effectively preventing contamination of a sample and performing quantitative analysis of an ultra-light element accurately without being affected by the contamination. As a means for achieving the above object, a cold trap is provided near the sample surface onto which an electron beam is irradiated. A concrete configuration of the sample contamination prevention apparatus according to the present invention will be described using FIGS. 1, 2, and 3.

Reference numeral 1 denotes a vacuum vessel housing an electron optical system, X-ray spectroscopy system, and observation optical system

(which are not shown), reference numeral 2 an evacuation pump of the vessel 1, reference numeral 3 a cold trap (hereinafter, referred to as "trap for main body"). A vessel 4 containing a coolant is suspended from the upper wall of the vessel 1 at the portion above the evacuation pump 2. Reference numeral 5 denotes an objective lens, and reference numeral 6 a sample.

Reference numeral 7 denotes a liquid reservoir. One end of the liquid reservoir 7 is, as shown in FIG. 2, closed by a lid 8 having a satisfactory heat transfer property. Liquid introduction pipes 9 and 10 are inserted from the other end side of the liquid reservoir 7 and are arranged in parallel to each other. The openings for the liquid introduction pipes 9 and 10 are air-tightly closed.

Reference numeral 11 denotes a cooling plate having an electron beam passing hole 11'. The cooling plate is provided near the sample surface and is fixed directly or through a heat transfer plate 12 to the lid 8 of the liquid reservoir 7. The components 7 to 11 constitute a cold trap (hereinafter, referred to as "cold trap for sample chamber").

The liquid reservoir having the above configuration is air-tightly attached to the side wall of the vacuum vessel 1 through a holding pipe 14 having a flange 13 and is inserted inside the vacuum vessel 1. Reference numeral 15 denotes a cover, which has a function of holding the liquid introduction pipes 9 and 10 as well as keeping airtight.

The lower side liquid introduction pipe 9 is inserted inside the reservoir more deeply than the upper side liquid introduction pipe 10. The other end of the lower side liquid introduction pipe 9 is connected to a container 16 for containing a coolant such as liquid nitrogen through a liquid feed pump P, and a liquid feed channel is formed such that liquid flows from the container 16, through the pump P, liquid introduction pipe 9, liquid reservoir 7, to the liquid introduction pipe 10.

The liquid feed pump P has a configuration as shown in FIG. 3. In FIG. 3, reference numerals 31, 32, and 33 denote coaxially-provided pipes. The upper end of the pipe 31 (hereinafter, referred to as "outside pipe") is tightly sealed to the pipe 32 (hereinafter, referred to as "intermediate pipe"), and the other end thereof is air-tightly connected to the liquid nitrogen container 16 through a cap 34. The one end of the intermediate pipe 32 is connected to a carburetor 35, one end of the pipe 33 (hereinafter, referred to as "axial pipe") is connected to the abovementioned liquid introduction pipe 9, and other ends of

the intermediate pipe 32 and axial pipe 33 open in the liquid nitrogen contained in the container 16.

Reference numeral 36 denotes a pressure rubber bulb fitted to the outside pipe 31 through a branch pipe 31', and reference numeral 37 is a valve provided at the connection portion between the branch pipe 31' and outside pipe 31.

Next, the action of the sample contamination prevention apparatus shown in FIG. 1 will be described, focusing the operation of the above pump mechanism.

When the pressure rubber bulb 36 is squeezed two or three times, before irradiation of an electron beam onto the sample 6, the liquid in the container 16 is pressurized by the pressure, with the result that the liquid nitrogen moves upward in the intermediate pipe 32 and axial pipe 33. The liquid nitrogen moving upward in the axial pipe 33 is guided to the liquid reservoir 7 by the liquid introduction pipe 9, and liquid nitrogen moving upward in the intermediate pipe 32 is guided to the carburetor 35. In this state, the squeeze of the pressure rubber bulb 36 is stopped.

Comment [s1]: 37→36

The liquid nitrogen supplied to the carburetor 35 is evaporated therein, and expansion pressure derived from vaporization pressurizes the liquid nitrogen through the intermediate pipe 32. With this pressure, the liquid nitrogen in the container 16 is always fed to the liquid reservoir 7 through the axial pipe 33 and liquid introduction pipe 9. When the liquid nitrogen in the liquid reservoir 7 exceeds a certain amount, it is discharged from the upper side liquid discharge pipe 10.

Comment [s2]: 36→35

As a result, the cooling plate 11 provided on the electron beam side at a portion near the sample is extremely cooled by heat transfer effect. Thus, oil vapor or carbon in the organic substance floating near the sample 6 is condensed and adheres to the cooling plate to be removed. Further, carbon heated by the electron beam irradiation and adhering to the electron beam irradiation surface of the sample is also condensed and adheres to the cooling plate to be removed by the existence of the extremely cooled cooling plate provided near the sample. Thus, it is possible to prevent contamination of the sample to the full extent.

FIG. 4 and tables 1, 2 show measurement results concerning contamination of samples (copper having a purity of 99.9%, and magnesium

having a purity of 99%) both in the cases where the contamination prevention apparatus according to the present invention shown in FIG. 1 is provided and where it is not provided. As is clear from the measurement results, in the case where the contamination prevention apparatus shown in FIG. 1 is provided, the increasing rate of contamination of the magnesium and increasing rate of contamination of the copper were 1/10 and 1/11 (relative to the case where the contamination prevention apparatus according to the present invention is not provided). Further, it can be understood that it is possible to obtain contamination prevention effect only with the trap for sample chamber (cold trap provided above the sample) without the trap 3 for main body provided above the evacuation pump as shown in FIG. 1.

In the embodiment shown in FIG. 1, the pump having the configuration shown in FIG. 3 is used as the liquid feed pump to automatically feed the liquid nitrogen to the liquid reservoir 7. Alternatively, however, a manual pump may be used to intermittently feed a coolant such that a certain amount of the coolant is stored in the liquid reservoir. Further, a configuration in which liquid introduction pipes are newly provided at the upper and lower portions of the liquid reservoir wherein the coolant is supplied through the lower liquid introduction pipe and coolant is discharged through the upper liquid introduction pipe may be employed. In this case, it is possible to confirm whether the certain amount of the coolant is stored in the liquid reservoir or not by simply confirming the amount of discharged liquid and, when the lower liquid introduction pipe for feeding the coolant is inserted all the way into the liquid reservoir, it is possible to effectively cool the cooling plate with a small amount of the coolant.

Further, although the liquid nitrogen is used as the coolant, any coolant may be used as long as it can cool the cooling plate to a temperature at which a sample contamination gas such as liquid helium existing in the vacuum vessel is condensed.

As described above in detail, the sample contamination prevention apparatus according to the present invention has a configuration in which the cooling plate is provided near the sample surface on the electron beam irradiation side, and coolant from the coolant container is supplied to the liquid reservoir provided with the cooling plate through a piping system by the liquid feed pump.

Thus, as is clear from the above-mentioned measurement results, it is possible to prevent contamination of the sample to the full extent. By providing the apparatus of the invention, analysis results excellent in reproducibility. Further, the configuration in which the coolant is supplied from the external container through the liquid feed pump system enables extended analysis.

While, in the X-ray micro-analyzer, an observation mechanism, a sample micro-movement mechanism, an X-ray detector, and the like are provided in the vacuum vessel housing the sample, the sample contamination prevention apparatus according to the present invention only has the liquid introduction pipes inserted thereinto, preventing other mechanism from being adversely affected and making the entire structure simple. When the apparatus is configured to be connected in a joint manner as denoted by the dotted line of FIG. 1, handling becomes easier.

[What is Claimed is:]

A sample contamination prevention apparatus in an X-ray micro-analyzer, the sample contamination prevention apparatus comprising:

a liquid reservoir 7 inserted from outside into a vacuum vessel 1 and air-tightly fixed thereto;

a cooling plate 11 connected to the liquid reservoir 7 and having an electron beam passing hole 11' near the portion above a sample 6;

a pair of liquid introduction pipes 9 and 10 inserted into the liquid reservoir 7; and

a pump mechanism P for feeding a coolant in a coolant container 16 to the liquid reservoir 7, wherein

one of the pipes 9 and 10 is used for feeding the coolant and the other one of them is used for discharge of the coolant.

□□□

□□□□□□

□□□□ X-ray intensity

□□□□ Measurement condition

□□□□ Acceleration voltage

□□□ X-ray diameter

□□□□ Measurement time

Translation of Utility Model Publication

Table -1 Measurement result concerning contamination of Mg (purity: 99%)

Trap type	Vacuum degree (mmHg)	Ckd at measurement start time (C.P.S)	Ckd at 4 minutes after start of measurement (C.P.S)	Ckd derived from contamination (C.P.S)	Fluctuation of chart sheet (cm)	contamination reduction rate
No trap	3.8×10^{-5}	150	710	560	13.5	1
Use only trap for sample chamber	3.5×10^{-5}	130	460	330	6.7	0.5
Use both trap for sample chamber and trap for main body	3.3×10^{-5}	120	200	80	1.8	0.13

Table -2

Trap type	Vacuum degree (mmHg)	Ckd at measurement start time (C.P.S)	Ckd at 4 minutes after start of measurement (C.P.S)	Ckd derived from contamination (C.P.S)	Fluctuation of chart sheet (cm)	contamination reduction rate
No trap	3.8×10^{-5}	250	970	720	14	1
Use only trap for sample chamber	3.5×10^{-5}	250	530	280	3.8	0.27
Use both trap for sample chamber and trap for main body	3.3×10^{-5}	250	320	70	1.3	0.093